

PATENT SPECIFICATION

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(54) WOOD PRESERVATION

(71) We, FOREST PRODUCTS UTILIZATION LABORATORY a Government agency of the State of Mississippi, United States of America, of State College, Mississippi 39762, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the pressure impregnation of wood with water-borne wood treatment materials to improve the properties of the wood. More particularly, this invention relates to a process for the impregnation of wood in which water-borne wood treatment materials are deposited within the wood, as by precipitation or chemical affixation, in a manner that combines the effectiveness and other advantages of the full-cell process with low residual liquids in the wood that can be obtained with empty-cell processes.

The present invention contemplates the use of what basically may be considered to be an "empty-cell" process as distinguished from a "full-cell" process. The essential difference between these two processes lies in the fact that in the full-cell process, the liquid forced into the wood is retained by the wood after impregnation. In contrast to this, in the empty-cell process, most of the treatment fluid is expelled from the wood after impregnation. The terms "full" and "empty" derive from the fact that the cells of the wood are substantially filled with impregnants in the full-cell process, but tend only to be coated with the impregnant in the empty cell process.

The full-cell process makes use of a vacuum/pressure impregnation cycle in which the wood is first placed under vacuum and then, without admitting air, the treatment vessel is filled with the treatment liquid. After the wood is fully immersed in the liquid, the pressure is increased to perhaps ten atmospheres or so and the liquid is forced into the wood. After the wood has been treated to refusal, or until a predetermined gross absorption of the treatment liquid has been achieved, the pressure is relieved and the treatment fluid is drained from the vessel. Usually a short vacuum cycle follows to remove excess fluid from the surface of the wood.

The distinctive feature of the empty-cell process is that at the end of the process, the pressure is reduced to, and preferably below, the pressure within the wood prior to impregnation so that a knockout of treatment liquid will result. The most common way to achieve this is to place the wood under pressure prior to the time that it is contacted with the treatment liquid. This initial pressure is maintained during the time that the treatment vessel is filled with the treatment liquid and then the pressure is increased to a second higher pressure, forcing the treatment liquid into the wood against the air pressure initially established within the wood. As a result, when the pressure is relieved, the air compressed within the wood expands and expels much of the liquid that was forced into the wood. Equivalent results can be obtained by starting the impregnation while the wood is

at or slightly below atmospheric pressure and then pulling a vacuum at the end of the treatment cycle. In any event, it is common to establish a vacuum in the treatment vessel after the impregnation pressure has been relieved to increase the recovery of the treatment liquid and shorten the period of time in which liquid will drip from the surface of the wood. The liquid expelled from the wood by the difference between the internal and external pressures is referred to as "kickout".

The empty-cell process is especially advantageous as compared with the full-cell process for treating wood with water-borne materials since the wood, after treatment, is ready for economic shipment, further treatment, or immediate use since there is no need to dry the wood to remove the treatment liquid, which may be toxic, from the interior surfaces of the wood.

Despite the inherent advantage of ending up with "dry" wood, the empty-cell process has not, for some purposes, been of practical utility in impregnating wood for a number of different reasons. For example, when treating wood with water-soluble preservatives (e.g., chromium/copper/arsenic salts known as "CCA salts"), the kickout that is inherent in empty-cell processes cannot economically be reused since the kickout will include water-soluble reducing substances leached from the wood (e.g., hemicelluloses), which, it is generally believed, react with the CCA salts to precipitate a dense sludge that fouls the equipment. This makes it impossible to recycle kickout without periodically removing insolubles, and further, the kickout cannot be discharged as waste because of its toxicity and the danger it poses to the environment. For these and other reasons, such as the preferential rate at which the CCA salts are extracted from solution, it has become standard practice to avoid problems attendant with recycling or otherwise disposing of depleted CCA solutions by using full-cell techniques and simply letting the treatment liquid remain in the wood until it evaporates which, if air dried, may take up to several months.

In addition to CCA salts, another effective and commonly used wood preservative that is difficult to use in empty cell processes is chlorinated phenol, most commonly the tetra and penta chlorophenols (collectively referred to as "PCP"). PCP is not soluble in neutral or acid solutions and, in order to form an impregnating solution, it is conventional to dissolve PCP in high-boiling petrochemicals, Stoddard's solvents or kerosene. Empty-cell impregnation methods are sometimes used to coat the cell walls with the preservative solution since the hydrocarbon solvents must be recovered to make the process economic and to prevent bleeding of the oil-based solution which would make it impossible to apply conventional finishes to the wood. However, even though most of the treatment solution is removed from the wood in empty-cell processes, the amount of solvent consumed represents the major portion of the cost of materials in impregnating the wood with PCP.

To reduce the cost of the oil-based solvents, other processes have been replacing the empty-cell ones in which the solvent is a low boiling liquid that may be recovered and reused. Typically, in these processes, low-boiling aliphatic hydrocarbon solvents for PCP are volatilized after impregnation, leaving a water-insoluble residue of PCP within the wood. These processes are not without attendant disadvantages which include the increased cost of treatment apparatus, the need to recover the gasified solvents, both for purposes of economy and environmental protection, and, as has been suggested in the literature, the protection afforded the wood is less lasting when volatile solvents are used.

A further disadvantage in the use of low-boiling hydrocarbon solvents is that the protection afforded the wood at its outer surface is sometimes less than satisfactory. It has been suggested that the reason for this lies in the fact that during volatilization, some of the PCP adjacent the surface of the wood is carried away and the amount of PCP left at this region is inadequate to protect the wood.

This invention provides an improved process for the pressure impregnation of wood; the process being a modified empty-cell process that may economically be used for preserving wood with common wood preservatives such as CCA salts and PCP.

More particularly the invention provides an improved process for the pressure-impregnation of wood with water-borne preservatives in which the retention of treatment materials within the wood is maximized and the retention of the liquids within the wood is minimized.

The process of this invention moreover provides improved surface protection of wood impregnated by PCP compared with a process in which low-boiling hydrocarbons are utilized as solvents. Briefly, the process of this invention

comprises pressure impregnating wood with a water-borne treatment material, detaining the impregnated wood under pressure for a sufficient time for the water-borne treatment materials to precipitate on or otherwise react with the wood, and then reducing the pressure to at least, and preferably below, the pressure within the wood immediately prior to impregnation so that a significant kickout can be obtained. By these means, the amount of treatment materials retained within the wood is maximized and the retention of the liquids within the wood is reduced to a minimum.

In the case of treatments with CCA salts, the process of this invention is of particular utility since the kickout is substantially depleted of metal ions and recovery or disposal problems are largely avoided. Further, the treated wood as discharged from the treatment vessel is substantially "dry" which permits immediate use, shipment, or even subsequent treatment, such as impregnation with creosote to meet requirements for marine applications.

This invention is based on the observation that the treatment materials can be reacted *in situ* during pressure impregnation which makes it possible to protect wood, in the case of PCP, without the use of hydrocarbon solvents. In the practice of this invention, sodium penta (or tetra) chlorophenate can be dissolved in an alkaline solution, the wood impregnated with the solution, and the solution detained within the wood without permitting kickout to occur, until the natural acidity of the wood causes precipitation of water-insoluble PCP within the wood. If the acidity of the wood is not sufficient to precipitate PCP at a useful rate, the wood may be first treated in an empty-cell process with an acid, such as acetic acid, prior to impregnation with the sodium pentachlorophenate solution. It is believed that by acidifying the pentachlorophenate to form the insoluble PCP precipitate *in situ* in the wood, the retention of the PCP and protection of the wood is made more lasting than is achieved when using either volatile or non-volatile petroleum-based solvents.

The process of the invention comprises placing the wood in a pressure treatment vessel at an initial elevated pressure; introducing an aqueous treatment liquid into the vessel at the elevated pressure; increasing the pressure within the vessel to above the initial pressure to impregnate the wood with the treatment liquid; maintaining the pressure within the vessel sufficient to retain the treatment liquid in the wood at a temperature of at least 65°C for a period of at least one hour after impregnation is complete until at least 90% of the treatment materials carried in the treatment liquid become affixed to or deposited within the wood, reducing the pressure within the vessel to or below the initial pressure to permit the expulsion of spent treatment liquid from the wood; and collecting the spent treatment liquid.

The reaction between the treatment materials and the wood is greatly accelerated when the wood is heated during the time the impregnating solution is held within the wood. Suitable times and temperatures will vary substantially, depending upon the species of wood being treated and the nature of the treatment liquid, but can easily be determined by simple experimentation.

By way of example, representative data has been obtained in treating Southern pine with CCA salts and it was found that temperatures ranging from about 65°C to 120°C are useful. At temperatures lower than these, the reaction rates are inconveniently slow, and at higher temperatures, damage may be done to the wood. A preferred temperature range for treating Southern pine with CCA salts is from about 82°C to 115°C and, more preferably still, from about 93°C to 110°C.

Treatment times will vary considerably and, depending on the temperature used in the treatment cycle, satisfactory results have been obtained using treatment times of from one to seven hours. Longer times can, of course, be used, but in the interest of productivity of the process, no purpose is served in prolonging the treatment time after the desired reactions have neared or reached completion.

In the Examples which follow all percentages are on a weight basis.

EXAMPLE 1

A charge of kiln-dry (15% moisture content) Southern pine tomato stakes and grape stakes consisting of thirty tomato stakes measuring 1"x1"x72" (1.25 cubic feet) and 15 grape stakes measuring 1-5/8"x1-5/8"x72" (1.65 cubic feet) was given a modified empty-cell treatment with a 2.0% (oxide basis) solution of CCA as follows:

An initial pressure of 10 p.s.i. air was introduced into the treatment vessel and held for five minutes. The vessel was then filled with the CCA solution without

relieving the initial pressure, and the pressure was increased to 100 p.s.i. in a forty-five minute period by increasing the pressure 10 p.s.i. at five minute intervals.

The excess preservative was removed while maintaining the cylinder pressure at 100 p.s.i.

5 The gross absorption of the CCA solution during the pressurization period was 35.9 lbs/cu.ft. 5

The charge was steamed 1-1/2 hours at 100 p.s.i. pressure within the maximum temperature of 116°C being reached in one hour and then held at this temperature for a half hour.

10 A kick-back sample taken at the end of the steaming cycle had a pH of 3.1. 10

The charge was exposed to a 26" Hg. vacuum for one hour before being removed and weighed to determine net solution retention. The net solution retention obtained during treatment was 10.22 lbs/cu.ft.

15 Analysis of the original treating solution and kick-back resulting from the treatment of this charge gave the following results: 15

Sample	pH	%CuO	%CrO ₃	%As ₂ O ₅	% Reduction of:		
					CuO	CrO ₃	As ₂ O ₅
Treatment solution	1.5	0.345	0.958	0.718	—	—	—
20 Kick-back	3.1	0.021	0.016	0.019	93.9	98.3	97.4

EXAMPLE 2

Precipitation of Cu and Cr from acid copper chromate in kiln-dry (20% moisture content) Southern pine by steaming before permitting the kick-back to occur.

25 A piece of kiln-dry Southern pine 6-1/2" in diameter and 18" long was impregnated as follows: 25

A. Preservative Solution:

30 A 2.38% solution (oxide basis) of acid copper chromate was prepared from a commercial (Celcure) concentrate of this preservative containing 3.84% copper sulfate (anhydrous), 5.01% sodium dichromate (anhydrous), and 0.20% chromic acid (anhydrous) by dilution with water. The pH of this fresh solution was 3.4. 30

B. Impregnation Cycle:

35 40 p.s.i. initial air was held five minutes, the treatment vessel was filled with the preservative solution at this pressure, and the pressure was then increased to 140 p.s.i. and held for two hours. 35

Excess preservative solution was drained from the vessel while maintaining the cylinder pressure at 140 p.s.i.

40 A kick-back sample was taken at the end of the pressure period but before starting the steaming by momentarily reducing the pressure slightly. The pH of this kick-back sample was 3.85. 40

The gross absorption of preservative during the pressure period was 29.29 lbs/cu.ft.

45 The sample was steamed at 100°C for three hours while maintaining 140 p.s.i. pressure on the cylinder. 45

A kick-back sample taken at the end of the steaming cycle had a pH of 5.45.

The sample was exposed to a 26" Hg. vacuum for one hour before being removed and weighed to determine net solution retention.

50 A sample of the drip, obtained during the final vacuum had a pH of 5.50. The net solution retention obtained during treatment was 9.09 lbs/cu.ft. so that the gross absorption was reduced by over 20 lbs/cu.ft. 50

A disc obtained from the middle of this sample after treatment was dried in an oven to determine its average moisture content. Its moisture content expressed as a per cent of oven-dry weight was 40.3%.

55 Analysis of the samples obtained during the treatment of this sample for Cu and Cr gave the results indicated in the table below. In this table, Sample 1 was the original treatment solution; Sample 2 was the kick-back after impregnation but before steaming; Sample 3 was the kick-back after impregnation and steaming; and Sample 4 was the drip from the wood during final vacuum. 55

	Sample	pH	% CuO	% Reduction of CuO	% CrO ₃	% Reduction of CrO ₃	
	1	3.40	0.717	—	1.661	—	
	2	3.85	0.628	12.41	1.527	8.07	
5	3	5.45	0.014	98.06	0.027	98.37	5
	4	5.50	0.025	96.58	0.009	99.46	

The disc obtained from this sample after treatment and steaming indicated complete penetration by the preservative.

EXAMPLE 3

10 In this example, the wood was given an empty-cell pretreatment with a 0.5% Ba(OH)₂ solution prior to the treatment with CCA to determine if the wood sugars could be precipitated in the wood so that they would not contaminate the kickout.

15 Analysis of the kickout after the CCA impregnation cycle showed that the reduction of the treatment solution in CuO was 100.00% and 99.56% in CrO₃ and As₂O₅.

Enough CCA concentrate was added to a portion of the kickout to bring its concentration up to 2.0% (oxide basis).

20 No precipitate occurred in this sample after two weeks' storage in the laboratory, indicating that the wood sugars were precipitated in the wood and did not, to any observable extent, contaminate the kick-back.

EXAMPLES 4—6

25 In Examples 4 through 6, samples of Southern pine wood were treated with CCA salts under varying process conditions, as shown in the following table. In this table, the column headed "Initial p.s.i." indicates the pressure to which the wood was exposed prior to impregnation. The column headed "Impregnation p.s.i." indicates the pressure that was established within the treatment vessel after the vessel had been filled with the treatment liquid. The "Steaming Temperature" was the ultimate temperature reached, over a period of about an hour, after the impregnation pressure was imposed. The "Holding Time" is the period of time that the vessel was maintained at the impregnation pressure and the steaming temperature.

30 The other columns indicate the percentage of the active ingredients in the initial treatment solution, the kick-back, and the percentage of the active ingredients that were retained within the wood. Note that in Examples 4 and 5 where the temperature of the treatment vessel was not increased above ambient, the deposition of the CCA salts did not begin to approach completion even after a holding time of as long as six hours. In contrast to this, Example 6 shows that at a steaming temperature of 99°C, well over 95% of the CCA salts were deposited in the wood after a holding time of only two hours.

Example	Initial psi	Impregna- tion psi	Steaming Temp.	Holding Time	Treatment Solution			
					% CuO	% CrO ₃	% As ₂ O ₅	
4	10	100	None	3 hrs.	0.382	1.010	0.718	
5	10	110	None	6 hrs.	0.331	0.962	0.692	
45 6	10	120	99°C	2 hrs.	0.314	0.904	0.819	45

Example	Kick-back			Retained Salts			
	% CuO	% CrO ₃	% As ₂ O ₅	% CuO	% CrO ₃	% As ₂ O ₅	
4	0.139	0.425	0.070	63.6	57.9	90.3	
5	0.096	0.338	0.170	71.1	64.8	75.5	
50 6	0.011	0.006	0.017	96.5	99.3	97.9	50

EXAMPLE 7

A piece of kiln-dry Southern pine 5-3/4" in diameter and 18" long was impregnated as follows:

55 A. Preservative solution: 1.50% technical-grade sodium pentachlorophenate in water. Original pH—10.3.

B. Impregnation cycle:

The wood was placed in a pressure treatment vessel and pressurized with air at 30 psi. Without relieving this pressure, the vessel was filled with the preservative solution and the air pressure was increased to 150 psi which was held for two hours.

The preservative was drained away from the wood while the cylinder pressure was maintained at 150 psi. Gross absorption of preservative during the pressure period was 32.63 lb/cu.ft.

The wood was then steamed for three hours at 100°C while maintaining 150 psi pressure on the cylinder. A kick-back sample taken by momentarily reducing the pressure at the end of the steaming period had a pH of 5.0. The wood was placed under a vacuum of 26" Hg. for one hour and was then removed from the cylinder and weighed. The net solution retention by gain in weight during treatment was 7.63 lb/ft³ which means that the amount of kickback expressed as a per cent of total gross absorption was 76.6%.

A sample of the drip recovered during the final vacuum had a pH of 5.15.

C. Analysis of borings for PCP:

Three increment cores were taken near midlength of the sample at 120° intervals around the circumference. The cores were zoned into 1/2" segments and assayed for PCP by the lime ignition method. The results of the analyses were:

Depth from Surface (inches)	PCP Content (lb/cu.ft.)
0.0—0.5	0.395
0.5—1.0	0.154
1.0—1.5	0.195
1.5—2.0	0.154

D. Analysis of the original treating solution and kick-back after steaming for PCP:

Samples of the original treating solution and kick-back were analyzed by the lime ignition method for PCP with results as follows:

	% PCP Content
Original treating solution	1.143
Kick-back after steaming	0.026

The percentage of reduction in PCP (i.e., retained in the wood) was 97.7%.

E. Test for fixation of PCP to the wood:

General procedure—A 3/4" long disc was cut from midlength of the treated pole section. A 3/4" wide strip was sawn from this disc so that the pith was in the center of the strip. The outer 3" on opposite sides of the pith in this strip was sectioned into 1/2" increments for the acid test and 1" increments for the pH determination.

Acid test procedure—Each block was splintered into 1/8 inch square pieces and covered with 50 ml of boiling distilled water. The mixture was shaken for thirty minutes and filtered through Whatman (Registered Trade Mark) #541 filter paper. 3 ml of the filtrate was placed in a test tube and dilute HCl was added to determine if any PCP precipitated upon addition of the HCl. (Note: The sensitivity of this method is 100 ppm).

Results of Acid Tests:

Sample Distance from Wood Surface (inches)	Precipitate:	
	Present very slight	Absent
0.0 to 0.5		
0.5 to 1.0		yes
1.0 to 1.5		yes
1.5 to 2.0		yes
2.0 to 2.5		yes
2.5 to 3.0		yes

F. Moisture content after treatment:

A disc 3/4" thick was sawn from the middle of the treated pole section and oven-dried to determine the average moisture content immediately after treatment. The moisture content of this disc was 38.6%.

EXAMPLE 8

In Example 7 the acidity of the wood was solely relied upon to reduce the pH of the treatment solution to precipitate PCP within the wood. In this Example the wood was first treated with acetic acid in a modified empty-cell process prior to impregnation with the pentachlorophenate solution to determine if the added acidity would make the process more efficient.

A sample of Southern pine wood was placed in a treatment vessel and placed under 10 psi air pressure. Without relieving this pressure, a 0.8 molar solution of glacial acetic acid was introduced into the vessel and the air pressure was increased to 120 psi and held for two hours. The acetic acid was drained away from the wood while maintaining 120 psi air pressure and the wood was then steamed for three hours at 99°C. The pressure was then relieved and a vacuum pulled for one hour.

Immediately following the above acid treatment of the wood, the identical process operations were repeated using a 4% alkaline solution of sodium pentachlorophenate as the impregnate.

After treatment with the PCP, the kickback was found to contain less than 0.008% PCP, thus indicating that over 99.8% of the PCP in the treatment solution was deposited in the wood. A test for leachable PCP as described in Example 1 above was run and the results were negative, indicating that the PCP precipitated in the wood could not be leached in observable quantities (i.e., greater than 100 ppm) by boiling the treated wood in water.

Borings were made in the treated wood and the amount of PCP deposited in the wood was determined at various depths by lime ignition:

	Sample	PCP (lb/ft ³)	
25	0.0—0.5"	1.1077	25
	0.5—1.0"	0.8462	
	1.0—1.5"	0.7918	
	1.5—2.0"	0.7663	

The above Examples 7 and 8 demonstrate that the chemically precipitated PCP is tightly held and is not leachable, except in trace amounts. It is at once obvious that by achieving this fixation of the PCP within the wood, the effectiveness of the wood preservative will be maintained over prolonged periods of time.

A secondary benefit which is gained in affixing the PCP within the wood lies in the fact that the potential danger of PCP to the environment is greatly reduced when the PCP is tightly held within the wood. In contrast to the results obtained through the practice of this invention, when conventional high-boiling petrochemical solvents are used and PCP is not precipitated in the wood but is held in solution by organic solvents, the PCP may leach from the wood over a period of time, depending upon such factors as the type of soil and other ground conditions to which the wood is exposed, changes in temperature, barometric pressure, humidity, and other ambient conditions.

EXAMPLE 9

A charge of air-dried (25% moisture) pine fence posts about 4"x8'6" in size were placed in a pressure vessel and an initial air pressure of 20 psi was introduced into the treatment vessel and held for about five minutes. The vessel was then filled with a CCA solution (see Table 1) without relieving the initial pressure, and the pressure was increased to 140 psi over a period of about 55 minutes. The temperature of the CCA impregnating solution was about 32°C and the gross absorption achieved was 31.4 lb/cu.ft.

When the required gross absorption had been obtained steam was admitted to the coils in the cylinder and the CCA solution in the cylinder was heated to 93°C within 30 minutes and this temperature was maintained for 60 minutes. No precipitation of CCA salts from the treatment solution was noted during this heating cycle.

At the end of the heating period, the CCA solution was removed from the cylinder without permitting the pressure on the system to change, following which the pressure on the system was released to collect the kickback separate from the CCA solution. The temperature of the kickback solution was 82°C. A final vacuum of 25 in. Hg. for 60 minutes followed after releasing the pressure on the system.

The wood was removed from the cylinder and weighed. It was determined that the net preservative solution retention was 7 lb/cu.ft.

Results of analyses performed on the CCA before treatment, after treatment and heating and on a sample of the kickback after heating are contained in Table 1:

TABLE 1
Metal Content (% oxide basis)

5	Solution	pH	CuO	CrO ₃	As ₂ O ₅	Total Salt	5
	CCA before treatment	1.92	0.304	0.870	0.643	1.817	
	CCA after treatment and heating	2.03	substantially the same as above				
	Kickback	4.18	0.008	0.017	0.022	0.047	

10 An increment core was taken from midlength of each post for analysis for Cu, Cr and As retention after treatment. The cores were cut into 1/2" segments and similar segments were combined for analysis. Results for these analyses are contained in Table 2:

TABLE 2
Retention of Metals—lb/cu.ft.
(Oxide Basis)

15	Depth from Surface (inches)	CuO	CrO ₃	As ₂ O ₅	Total Salt	15
	0.0 to 0.5	0.161	0.352	0.251	0.765	
20	0.5 to 1.0	0.123	0.244	0.019	0.385	20
	1.0 to 1.5	0.074	0.177	0.014	0.265	

25 The modified empty-cell process of this invention has been described primarily with regard to the impregnation of wood with sodium chlorinated phenates and CCA salts, but it can readily be understood by those of ordinary skill in the wood treatment arts that the invention is of utility in treating wood with other wood conditioners. For example, the water-borne materials may include such things as wood-softening agents, antichecking agents, film-formers, coloring agents, flame retardants, antistatic agents, dimensional stabilizers, polymerizable mixtures and additional wood preservatives. The process of this invention may also be used to leach materials, such as sugars, from wood, or, conversely, to precipitate sugars in the wood prior to a subsequent preservation step. The pH of the treatment liquid can be adjusted to maximize solubilization of the sugars and, if it is the object to fix the sugars in the wood, cations, such as barium or copper, may be included in the treatment liquid to form insoluble products with the sugars.

35 The modified empty-cell process of this invention makes it possible to conduct a plurality of sequential impregnations without the intervening drying or curing steps required in the full-cell process. For example, substantially immediately after wood is treated with CCA salts in accordance with this invention, it may be impregnated with an oil-based preservative. Another example would be to follow the CCA treatment with a PCP treatment. This increases the degree of protection for the wood and is advantageous if arsenic salts must be excluded from the treatment liquid because of environmental hazards.

45 In a further modification of this invention, CCA salts can be deposited at the surface regions of wood while the wood is being treated with a solution of PCP and liquified hydrocarbon. When the pressure is relieved on the system, volatilization of the hydrocarbon will cause knockout of the CCA treatment liquid.

50 In the above examples, the treatment solution was removed from contact with the wood (while maintaining impregnation pressures) prior to the time that the wood was heated. This is not essential and, if desired, the temperature of the wood may be raised by heating the impregnating solution (as by steam coils) prior to the time it is drained from the vessel. In another variation of the practice of this invention, the treatment solution may, in some instances, be heated prior to the time it is introduced into the vessel. Also, the treatment liquid may be drained from the vessel while maintaining sufficient pressure to avoid knockout, and steam or a second liquid used to heat the wood.

55 In this latter regard, a liquid may be introduced into the vessel while maintaining sufficient pressure to prevent knockout. If a hot water bath is used for

5 this final treatment, it may prove advantageous to include water-borne wood
treatment materials such as the above-mentioned wood-softening agents,
anticlecking agents, film-formers, coloring agents, flame retardants, antistatic
agents, dimensional stabilizers, and other wood-treating agents. Also, if the first
10 treatment was with PCP, a dilute solution of CCA salts may advantageously be
included in the water to provide additional protection to the surface of the wood.
Since the CCA salts will tightly adhere to the wood, this additional protection may
prove to be of value to protect against any possible loss or migration of the PCP
from the surface areas of the wood. Thus, it can be understood that this final
15 treatment step may serve not only to aid in precipitating or affixing the preservative
to the wood, but also to improve at least the surface properties of the wood.

WHAT WE CLAIM IS:—

- 15 1. A method for the treatment of wood comprising:
placing the wood in a pressure treatment vessel at an initial elevated pressure;
introducing an aqueous treatment liquid into the vessel at the elevated
pressure;
increasing the pressure within the vessel to above the initial pressure to
impregnate the wood with the treatment liquids;
20 maintaining the pressure within the vessel sufficient to retain the treatment
liquid in the wood at a temperature of at least 65°C for a period of at least one hour
after impregnation is complete until at least 90% of the treatment materials carried
in the treatment liquid become affixed to or deposited within the wood;
reducing the pressure within the vessel to or below the initial pressure to
25 permit the expulsion of spent treatment liquid from the wood; and
collecting the spent treatment liquid.
2. A method according to Claim 1 wherein the aqueous treatment fluid
contains a wood preservative.
3. A method according to Claim 2, wherein the wood preservative contains
salts of copper, chromium or arsenic.
- 30 4. A method according to Claim 2, wherein the wood preservative is a sodium
chlorophenate.
5. A method according to Claim 4, wherein the aqueous treatment fluid is an
alkaline solution of sodium pentachlorophenate.
- 35 6. A method according to any one of Claims 1 to 5, wherein the wood is heated
to a temperature within the range from 65°C to 120°C.
7. A method according to any one of Claims 1 to 6, wherein the wood is heated
by heating the treatment fluid.
8. A method according to any one of Claims 1 to 6, wherein after impregnation
is complete excess treatment liquid is drained from the vessel.
- 40 9. A method according to Claim 8, wherein the wood is heated by steam.
10. A method according to Claim 8 or Claim 9, wherein the wood is heated
with a heated second treatment liquid.
11. A method according to Claim 10, wherein the second treatment liquid
contains wood treatment agents selected from the group consisting of coloring
45 agents, wood-softening agents, anticlecking agents, film formers, flame retardants,
antistatic agents, polymerizable mixtures and additional wood preservatives.
12. A method according to any one of Claims 4 to 11, wherein the wood is
pretreated with an acid.
13. A method according to Claim 12, wherein the acid is acetic acid.
- 50 14. A method according to any of the preceding claims wherein the wood is
pretreated using the method of Claim 1 with hot water or steam as the aqueous
treatment liquid.
15. A method according to any of the preceding claims wherein the wood is
pretreated with metal ions to precipitate sugars within the wood.
- 55 16. A method according to Claim 1, wherein the wood is impregnated with a
solution of pentachlorophenol and a low-boiling hydrocarbon or chlorinated
solvent for the pentachlorophenol before the aqueous treatment liquid is
introduced into the vessel and wherein the aqueous treatment liquid contains CCA
salts.
- 60 17. A method according to any one of Claims 1 to 16, wherein the treated
wood is further impregnated with creosote.

18. A method as claimed in Claim 1 conducted substantially as described in any of Examples 1 to 3 and 6 to 9 herein.

19. Impregnated wood whenever obtained by a method as claimed in any of Claims 1 to 18.

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